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IS 15929 (2012): Pectin, Food Grade - Specification [FAD 8: Food Additives]

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भारतीय मानक

पेक्टीन, खाद्य ग्रेड — विशिष्टि

Indian Standard

PECTIN, FOOD GRADE — SPECIFICATION

ICS 67.220.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 2012 **Price Group 3**

Food Additives Sectional Committee, FAD 8

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Pectin is used as diluent or filler material in synthetic food colour preparations. It is also used as gelling agent, thickener, stabilizer and emulsifier. The main raw-materials for pectin production are dried citrus peel or apple pomace, both by-products of juice production. Pomace from sugar-beet is also used to a small extent. From these materials, pectin is extracted by adding hot dilute acid at *p*H values from 1.5-3.5. During several hours of extraction, the protopectin loses some of its branching and chain length and goes into solution. After filtering, the extract is concentrated in vacuum and the pectin then precipitated by adding ethanol or isopropanol. Precipitated pectin is then separated, washed and dried. Treating the initial pectin with dilute acid leads to low esterified pectins. When this process includes ammonium hydroxide, amidated pectins are obtained. After drying and milling pectin is usually standardized with sugar and sometimes calcium-salts or organic acids to have optimum performance in a particular application.

To ensure the quality of the product, it is necessary to have quality control specifications. This standard, which is based on Joint FAO/WHO Expert Committee on Food Additives (JECFA) standard for pectin has been formulated to provide the necessary guidelines to manufacturers and the consumers for maintaining quality control.

In the formulation of this standard, due consideration has been given to the *Food Safety and Standards Act,* 2006 and the *Rules* framed thereunder. Due consideration has also been given to the *Legal Metrology (Packaged Commodities)* Rules*,* 2011. However, this standard is subject to restrictions imposed under these Rules, wherever applicable.

Description

Common name — Pectin
C.A.S. number — 9000-69-5

In reporting the results of a test or analysis made in accordance with this standard, if the final value observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PECTIN, FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and test for pectin, food grade.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1699 : 1995	Methods of sampling and test for food colours (*second revision*)
2491 : 1998	Food hygiene — General principles — Code of practice (*second revision*)
5194 : 1969	Method for determination of nitrogen — Kjeldahl method

3 REQUIREMENTS

3.1 Description

Pectin is available as white, yellowish, light greyish or light brownish powder. It consists mainly of the partial methyl esters of polygalacturonic acid and their sodium, potassium, calcium and ammonium salts; obtained by extraction in an aqueous medium of appropriate edible plant material, usually citrus fruits or apples; no organic precipitants shall be used other than methanol, ethanol and isopropanol; in some types a portion of the methyl esters may have been converted to primary amides by treatment with ammonia under alkaline conditions. Sulphur dioxide may be added as a preservative. The commercial product is normally diluted with sugar for standardization purposes. In addition to sugars, pectins may be mixed with suitable food grade buffer salts required for *p*H control and desirable setting characteristics. The article of commerce may be further specified by *p*H value, gel strength, viscosity, degree of esterification and setting characteristics.

3.2 Test for Pectins

It should pass the test when tested in accordance with Annex A.

3.3 Test for Amide Group (for Amidated Pectins Only)

It should pass the test when tested in accordance with Annex B.

3.4 The material shall also conform to the requirements given in Table 1.

Table 1 Requirements for Pectins, Food Grade

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Annex of this Standard	Clause of Other Standards
(1)	(2)	(3)	(4)	(5)
i)	Loss on drying, percent by mass, *Max*	12	C	—
ii)	Sulphur dioxide, mg/kg, *Max*	50	D	—
iii)	Lead (as Pb), mg/kg, *Max*	2	—	15 of IS 1699
iv)	Copper (as Cu), mg/kg, *Max*	300	—	15 of IS 1699
v)	Arsenic (as As), mg/kg, *Max*	5	—	15 of IS 1699
vi)	Methanol, percent by mass, *Max*	1	E	—
vii)	Ethanol, percent by mass, *Max*	1	E	—
viii)	2-propanol, percent by mass, *Max*	1	E	—
ix)	Methanol, ethanol and 2-propanol, percent by mass, *Max*	1	E	—
x)	Acid insoluble ash, percent by mass, *Max*	1	F	—
xi)	Total insolubles, percent by mass, *Max*	3	G	—
xii)	Nitrogen, percent by mass, *Max*	2.5	—	IS 5194
xiii)	Galacturonic acid, percent by mass on ash-free and dried basis, *Min*	65	H	—
xiv)	Degree of amidation, percent by mass of total carboxyl groups of pectin, *Max*	25	H	—

3.5 The material shall be processed, packed, stored and distributed under hygienic conditions in licensed premises (*see* IS 2491).

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be securely packed in well filled containers with minimum access to air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

4.3 Marking

Each container shall be legibly and indelibly marked with the following information:

a) Name of the material including the words 'Food Grade';
b) Name and address of the manufacturer;
c) Batch or Code number;
d) Net quantity when packed;
e) Instruction for storage;
f) Best before date (month and year to be given by the manufacturer); and
g) Any other requirements as given under the *Legal Metrology (Packaged Commodities) Rules,* 2011 and *Food Safety and Standards (Packaging and Labelling) Regulation,* 2011.

4.3.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

4.3.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representatives samples of the material shall be drawn according to the method prescribed in **4** of IS 1699.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of test or analysis.

ANNEX A

(*Clause* 3.2)

TEST FOR PECTINS

A-1 PROCEDURE

Moisten 0.05 g of the sample with 2-propanol. Add 50 ml of water on a magnetic stirrer. Adjust *p*H to 12 using 0.5 mol/l sodium hydroxide and let the solution remain without stirring for 15 min. Reduce *p*H to 7.0 with 0.5 mol/l hydrochloric acid. Adjust to 100.0 ml with water. Make up samples in 1 cm quartz cuvettes as follows:

	*Buffer p*H 7.0 (*see* Note 1)	*Sample Solution* ml	*Water* ml	*Enzyme* (*see* Note 2)
Enzyme blank	0.5	1.0	1.0	—
Sample blank	0.5	—	1.5	0.5
Sample	0.5	1.0	0.5	0.5

NOTES

1 Dissolve 6.055 g of tris (hydroxymethyl) aminomethane and 0.147 g of calcium chloride dihydrate in water to 1 l. Adjust *p*H to 7.0 with 1 mol/l hydrochloric acid.

2 Dilute pure pectate lyase 1:100 with buffer *p*H 7.0.

Shake the solutions well, and measure the absorbance at 235 nm at 0 and 10 min.

A-2 CALCULATIONS

A_0 = absorbance at 0 min = Sample – (enzyme blank + sample blank)

A_{10} = absorbance at 10 min = Sample – (enzyme blank + sample blank)

The amount of unsaturated product produced is proportional to the change in absorbance ($A_{10} - A_0$). This value should be greater than 0.023. This distinguishes pectins from other gums, which show essentially no change.

ANNEX B

(*Clause* 3.3)

TEST FOR AMIDE GROUP (FOR AMIDATED PECTINS ONLY)

B-1 PROCEDURE

Add 2 ml of hydrochloric acid and 50 ml of 60 percent ethanol to 0.5 g of the sample, and stir well for 20 min. Transfer to a fritted glass filter tube wash with six 10 ml portions of the HCl 60 percent ethanol mixture. Dissolve in 100 ml distilled water; it may be necessary to add a few drops 0.1 mol/l sodium hydroxide to achieve solution. Transfer 4 ml of this solution into a test tube (recommended dimensions 15.5 mm inner diameter and 146 mm length). Add 1 ml 5 mol/l sodium hydroxide and mix. The mixture will form a gel. Fill a small glass tube (recommended dimensions 7.8 mm inner diameter and 79 mm length) with 2.5 ml boric acid TS and let glide into the test tube. Close with parafilm and incubate overnight at 30º. In case of presence of amide groups the indicator changes its colour from red to green, due to release of ammonia.

ANNEX C

[*Table* 1, *Sl No.* (i)]

TEST FOR LOSS ON DRYING

C-1 PROCEDURE

Mix the sample well and accurately weigh 1 g of the substance. Reduce the sample to a fine powder. Tare a glass stoppered, shallow weighing bottle that has been dried for 30 min at 105ºC for 2 h. Transfer the sample into the bottle, replace the cover, and weigh the bottle and the sample. Distribute the sample as evenly as practicable to a depth of about 5 mm. Place the bottle with its contents in the drying chamber, removing the stopper and leaving it also in the chamber and dry the sample at 105ºC for 2 h. Upon opening the chamber, close the bottle promptly and allow it to come to room temperature in a desiccator before weighing.

NOTE — Suitable precautionary steps should be taken when weighing hygroscopic or deliquescent samples to ensure that they do not absorb moisture.

ANNEX D

[*Table* 1, *Sl No.* (ii)]

TEST FOR SULPHUR DIOXIDE

D-1 PROCEDURE

Suspend 100 g of the sample in 500 ml of methanol in a 1 000-ml round bottom flask, which is provided with a gas inlet tube reaching almost the bottom and connected to the neck with a reflux condenser. Prepare a glass joint connection from the condenser to an absorption flask or U-tube containing 10 ml of 3 percent hydrogen peroxide solution neutralized to methyl red TS. Connect the gas inlet tube with an oxygen free source of carbon dioxide or nitrogen, and maintain a gas stream so as to cause steady bubbling. As soon as the apparatus is flushed free of air, pour 30 ml of hydrochloric acid solution (10 ml concentrated HCl + 20 ml H_2O) into the reflux condenser, and immediately connect the absorption flask or U-tube. Heat slowly until methanol starts refluxing, and reflux gently for 2 h. Disconnect the apparatus and titrate the hydrogen peroxide solution against methyl red TS with 0.01 mol/l sodium hydroxide. Each ml of 0.01 mol/l sodium hydroxide corresponds to 0.32 mg of SO_2.

ANNEX E

[*Table* 1, *Sl No.* (vi), (vii), (viii) *and* (ix)]

DETERMINATION OF METHANOL, ETHANOL AND 2-PROPANOL CONTENT

E-1 PROCEDURE

Weigh 200 mg of urea into a 25-ml amber glass vial. Purge with nitrogen for 5 min and then add 1 ml of saturated oxalic acid solution, close with a rubber stopper and swirl. Add 1 ml of a sample solution (100 mg of sample in 10 ml of water using sodium chloride as a dispersing agent,m if necessary), 1 ml of internal standard solution (50 mg *n*-propanol in 1 000 ml of water) and simultaneously start a stop watch ($T = 0$). Swirl the vial and recap with an open screw cap fitted with a silicone rubber septum. Swirl until $T = 30$ s. At $T = 45$ s inject through the septum 0.5 ml of an aqueous solution of sodium nitrite (250 g/l). This will convert the alcohols to their corresponding nitrite esters. Swirl until $T = 70$ s and at $T = 150$ s, withdraw through the septum 1.0 ml of the headspace using a pressure lock syringe. Insert syringe needle in the injection port; pre-compress the sample, then open the syringe and inject the sample. Use the following conditions:

a) Column : length: 90 cm, diameter: 4 mm, material: glass
b) Packing : First 15 cm packed with chrompack (or equivalent) and the remainder with Porapak R 120-150 mesh (or equivalent)
c) Carrier gas : Nitrogen
d) Flow rate : 80 ml/min
e) Detector type : FID
f) Temperatures : injection port: 250°C, column: 150° isothermal

Quantify the methanol, ethanol and 2-propanol present in the sample by comparing the peak areas with the corresponding peaks obtained by chromatographing the headspace produced by substituting in the procedure 1 ml of an aqueous solution containing 50 mg/l each of methanol, ethanol and 2-propanol for 1 ml of sample solution.

ANNEX F

[*Table* 1, *Sl No.* (x)]

TEST FOR ACID INSOLUBLE ASH

F-1 PROCEDURE

Accurately weigh a known quantity of sample (depending on the ash content such that about 20 mg of ash is obtained) in a tared crucible, ignite at a low temperature (about 550°), not to exceed a very dull redness, until free from carbon, cool in a desiccator, and weigh. If a carbon free ash is not obtained, wet the charred mass with hot water, spread the residue using a glass rod, dry it in an air oven and reignite. If a carbon-free ash is still not obtained, cool the crucible, add 15 ml of ethanol, break up the ash with a glass rod, then burn off the ethanol, again heat the whole to dull redness, cool in a desiccator. Boil the ash with 25 ml of dilute hydrochloric acid TS for 5 min, collect the insoluble matter on a suitable ash-less filter, wash with hot water, ignite at 800 ± 25°, cool, and weigh. Calculate the percentage of acid-insoluble ash from the weight of the sample taken.

NOTE — If difficulty with oxidizing organic material is found, the use of an ash aid such as ammonium nitrate may prove to be more satisfactory. Addition of a few drops of hydrogen peroxide facilitate oxidation of organic matter.

ANNEX G

[*Table* 1, *Sl No.* (xi)]

DETERMINATION OF TOTAL INSOLUBLES

G-1 PROCEDURE

Dry a 70 mm glass fiber filter paper (GF/B) (Whatman Code 1821 070) in an oven with fan set at 105ºC for about 1 h. Transfer the filter paper to a desiccator containing silica gel and allow to cool. Weigh the paper (M_1). Weigh about 1 g (S) of the sample into a 250 ml beaker. Add 5 ml of 2-propanol to disperse the sample. While stirring magnetically, add 100 ml of 0.03 mol/l sodium hydroxide containing 0.1 percent (*w/w*) ethylene diamine tetra acetic acid (Na salt), which has been filtered through GF/B paper. Stir for about 30 min at room temperature, then heat to boiling (remove heat, if excessive foaming occurs). Filter the hot solution through the glass fiber paper under vacuum using, for example a vacuum filtration kit with 3 piece Hartley funnel (70 cm), with heat resistant plate. Rinse the beaker five times and filter the rinsings with 100 ml of warm (about 50º) water that has been filtered through GF/B paper.

Dry the filter paper with the residue at 105º for 1 h. Transfer to desiccator containing silica gel and leave to cool. Weigh the paper (M_2). Calculate the percentage of total insolubles from

$$\text{Total insolubles (percent)} = [(M_2 - M_1)/S] \times 100$$

ANNEX H

[*Table* 1, *Sl No.* (xiii) *and* (xiv)]

TEST FOR GALACTURONIC ACID AND DEGREE OF AMIDATION

H-1 PROCEDURE

Weigh 5 g of the sample to the nearest 0.1 mg, and transfer to a suitable beaker. Stir for 10 min with a mixture of 5 ml of hydrochloric acid TS, and 100 ml of 60 percent ethanol. Transfer to a fritted glass filter tube (30 to 60 ml capacity) and wash with six 15-ml portions of the HCl 60 percent ethanol mixture, followed by 60 percent ethanol until the filtrate is free of chlorides. Finally wash with 20 ml of ethanol, dry for 2.5 h in an oven at 105º, cool and weigh. Transfer exactly one-tenth of the total net weight of the dried sample (representing 0.5 g of the original unwashed sample) to a 250-ml conical flask and moisten the sample with 2 ml of ethanol TS. Add 100 ml of solution is formed. Add 5 drops of phenolphthalein TS, titrate with 0.1 mol/l sodium hydroxide and record the results as the initial titre (V_1).

Add exactly 20 ml of 0.5 mol/l sodium hydroxide TS, stopper, shake vigorously and let stand for 15 min. Add exactly 20 ml of 0.5 mol/l hydrochloric acid and shake until the pink colour disappears. Titrate with 0.1 mol/l sodium hydroxide to a faint pink colour which persists after vigorous shaking; record this value as the saponification titre (V_2).

Quantitatively transfer the contents of the conical flask into a 500-ml distillation flask fitted with a Kjeldahl trap and a water-cooled condenser, the delivery tube of which extends well beneath the surface of a mixture of 150 ml of carbon dioxide free water and 20.0 ml of 0.1 mol/l hydrochloric acid in a receiving flask. To the distillation flask add 20 ml of a 1-in-10 sodium hydroxide solution, seal the connections, and then begin heating carefully to avoid excessive foaming. Continue heating until 80-120 ml of distillate has been collected. Add a few drops of methyl red TS to the receiving flask, and titrate the excess acid with 0.1 mol/l sodium hydroxide recording the volume required, in ml, as S. Perform a blank determination on 20.0 ml of 0.1 mol/l hydrochloric acid, and record the volume required, in ml, as B. The amide titre is ($B - S$).

Transfer exactly one-tenth of total net weight of the dried sample (representing 0.5 g of the original unwashed sample) and wet with about 2 ml ethanol in a 50-ml beaker. Dissolve the pectin in 25 ml 0.125 mol/*L* sodium hydroxide. Let the solution stand for 1 h with agitation at room temperature. Transfer quantitatively the saponified pectin solution to a 50-ml measuring flask and dilute to the mark with distilled water. Transfer 20.00 ml of the diluted pectin solution to a distillation apparatus and add 20 ml of Clark’s solution, which consists of 100 g of magnesium sulphate heptahydrate and 0.8 ml of concentrated sulphuric acid and distilled water to a total of 180 ml. This apparatus consists of a steam generator connected to a round-bottom flask to which a condenser is attached. Both steam generator and round-bottom flask are equipped with heating mantles.

Start the distillation by heating the round bottom flask containing the sample. Collect the first 15 ml of distillate separately in a measuring cylinder. Then start the steam supply and continue distillation until 150 ml of distillate have been collected in a 200-ml beaker. Add quantitatively the first 15 ml distillate and titrate with 0.05 mol/*L* sodium hydroxide to *p*H 8.5 and record volume required, in ml, as A.

Perform a blank determination on 20 ml distilled water. Record the required volume, in ml, as A_0. The acetate ester titre is $(A - A_0)$.

Calculate degree of amidation (as percent of total carboxyl groups) by the following formula:

$$100 \times \frac{B - S}{V_1 + V_2 + (B - S) - (A - A_0)}$$

Calculate in mg, of galacturonic acid by the following formula:

$$19.41 \times [V_1 + V_2 + (B - S) - (A - A_0)]$$

The mg of galacturonic acid obtained in this way is the content of one-tenth of the weight of the washed and dried sample. To calculate percent galacturonic acid on a moisture and ash free basis, multiply the number of mg obtained by 1 000/*x*, *x* being the weight, in mg, of the washed and dried sample.

NOTES

1 If the pectin is known to be of the nonamidated type, only V_1 and V_2 need to be determined and $(B - S)$ may be regarded as zero.

2 For pectins from apple or citrus $(A - A_0)$ is usually insignificant in calculating galacturonic acid and degree of amidation.

3 If desired, calculate degree of esterification (as percent of total carboxyl groups) by the following formula:

$$100 \times \frac{V_2 - (A - A_0)}{V_1 + V_2 + (B - S) - (A - A_0)}$$

4 If desired, calculate degree of acetate ester (as percent of total carboxylic groups from galacturonic acid) by the following formula:

$$100 \times \frac{(A - A_0)}{V_1 + V_2 + (B - S) - (A - A_0)}$$

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